ANSWER 1 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN GΙ

Ozonolysis of the pyrrolidinediones I (R = Ph, 4-ClC6H4; R1 = :O) afforded AB the pyrrolidinetriones, which in the presence of Lewis acids were converted into maleimide derivative Analogously, ozonolysis of the pyrrolidinones I (same R; R1 = OH) gave the pyrrolidinediones, which were converted into the pyridinetriones II (same R) via Lewis acid catalyzed isomerization to yield the trihydroxypyridones and ensuing air oxidation In solution two tautomeric forms of the pyridinetriones II may exist, both of which represent hydroxy azabenzoquiones. In two steps compds. II were transformed into azaquinone derivs. III. Representatives of another type of azaquinones are compds. IV (R1 = CO2Me, H). The azaquinone IV (R1 = CO2Me) reacted easily with acidic compds. or with 2-butenal. IT

548736-16-9P 548736-21-6P 548736-45-4P

548736-47-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation of azabenzoquinones by ring-expansion reactions)

RN548736-16-9 CAPLUS

2,3,6(1H)-Pyridinetrione, 4-hydroxy-5-phenyl- (9CI) (CA INDEX NAME) CN

RN 548736-21-6 CAPLUS

2,3,6(1H)-Pyridinetrione, 4-methoxy-5-phenyl- (9CI) (CA INDEX NAME) CN

RN 548736-45-4 CAPLUS

CN 2,5-Pyridinedione, 1,6-dihydro-6-hydroxy-4-methoxy-6-phenyl- (9CI) (CA INDEX NAME)

RN 548736-47-6 CAPLUS

CN 2,5-Pyridinedione, 6-(acetyloxy)-1,6-dihydro-4-methoxy-6-phenyl- (9CI) (CA INDEX NAME)

ACCESSION NUMBER:

2003:195519 CAPLUS

DOCUMENT NUMBER:

139:69126

TITLE: AUTHOR(S):

New azabenzoquinones by ring-expansion reactions Poschenrieder, H.; Stachel, H.-D.; Wiesend, B.;

Polborn, K.

CORPORATE SOURCE:

Department Pharmazie / Zentrum fur Pharmaforschung,

Universitat Munchen, Munchen, D 81377, Germany

SOURCE: Journal of Heterocyclic Chemistry (2003), 40(1), 61-69

CODEN: JHTCAD; ISSN: 0022-152X

PUBLISHER:

HeteroCorporation

DOCUMENT TYPE:

Journal

LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 139:69126

REFERENCE COUNT:

THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 2 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN GI

$$X \longrightarrow OR^2$$
 $N \longrightarrow R^1$

Ι

$$C1$$
 OH N Me O II

Title compds. I [R1 = H, alk(en/yn)yl, cycloalkyl, cycloalkenyl, aryl, aralkyl, heterocyclyl, heterocyclylalkyl; R2 = H, alk(en/yn)yl, cycloalkyl, cycloalkenyl, aryl, aralkyl, acyl, heterocyclyl, heterocyclylalkyl; W, X = H, halo] were prepared Dichloroacetyl chloride was reacted with N-methylformamide at reflux for a couple of hours. The crude reaction mixture was mixed with NaHCO3 and the product isolated by continuous extraction with ether and II was isolated and purified by distillation in

vacuo in 40% yield. Addnl. expts. showed the isolation of intermediates in the process, e.g., (Z)-2,3-dichloro-4-(N-formyl-N-methylamino)-4-oxobut-2-enoic acid and 3,4-dichloro-2-(N-methylformamido)-5-dichloroacetoxyfuran (characterized). II at 400 mg/kg (mice) showed the following tumor growth inhibition/tumor/cancer: 92%/MAC 13/colon, 97%/M5076/ovarian and 81-86%/MAC15/colon. I are anti-proliferative agents, especially tumor growth inhibitors and anti-cancer agents, antibiotics and/or antiviral agents.

IT 458523-68-7P

> RL: PAC (Pharmacological activity); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)

(antiproliferative agents)

RN458523-68-7 CAPLUS

2,5-Pyridinedione, 3,4-dichloro-1,6-dihydro-6-hydroxy-1-methyl- (9CI) CN(CA INDEX NAME)

ACCESSION NUMBER:

2002:716252 CAPLUS

DOCUMENT NUMBER:

137:232564

TITLE:

Preparation of dioxodihydropyridine derivatives as

antiproliferative agents

INVENTOR (S):

Ayuko, Washington Odur; Tisdale, Michael John;

Lattmann, Eric

PATENT ASSIGNEE(S):

EPX Research Limited, UK

SOURCE:

PCT Int. Appl., 34 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002072553	A1	20020919	WO 2002-GB1119	20020312

```
AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
             UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU,
             TJ, TM
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,
             CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
             BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
     GB 2373246
                          Α1
                                 20020918
                                             GB 2001-6137
                                                                    20010313
     CA 2441001
                          AA
                                 20020919
                                             CA 2002-2441001
                                                                    20020312
     EP 1377551
                          Α1
                                 20040107
                                             EP 2002-704961
                                                                    20020312
             AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
     BR 2002008101
                          Α
                                 20040302
                                             BR 2002-8101
                                                                    20020312
     US 2004106648
                          A1
                                 20040603
                                             US 2003-662555
                                                                    20030915
PRIORITY APPLN. INFO.:
                                             GB 2001-6137
                                                                    20010313
                                                                 Α
                                             WO 2002-GB1119
                                                                    20020312
OTHER SOURCE(S):
                         MARPAT 137:232564
REFERENCE COUNT:
                         10
                                THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS
                                RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
L_3
     ANSWER 3 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN
GI
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- * STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY AVAILABLE VIA OFFLINE PRINT *
- AB The title photog. material has ≥1 layer containing compound I-V (X1 = 0, S, SO2, NR1(R1 = H, alkyl, aryl, heterocyclyl); L1-15 = methine; n1-5 = 0-1; Z = non-metallic atoms required to form aromatic ring; Y = alkylsulfonyl, arylsulfonyl, aryloxycarbonyl, carbamoyl; R2 = aryl; R3 = R1; when R3 = aryl, R4 = aryloxycarbonyl, acylamino, ureido, carboxy, carbamoyl, cyano, hydroxy, alkoxy, aryloxy, amino, sulfamoyl, sulfone amido; when R3 = H, alkyl, heterocyclyl, R4 = H, alkyl, aryl, heterocyclyl, alkoxycarbonyl or group defined above for R4; R5-7 = organic group; X2-4 = O, S; R8, R9 = H, alkyl, aryl, heterocyclyl; each compound of I-V containing at least 1 of carboxy, sulfone amido, or sulfamoyl).

ΙT 167014-99-5

> RL: RCT (Reactant); RACT (Reactant or reagent) (preparing specific compound for photog. material)

167014-99-5 CAPLUS RN

CN 3-Pyridinecarbonitrile, 1,2,5,6-tetrahydro-6-hydroxy-4-methyl-2,5-dioxo-(CA INDEX NAME)

ACCESSION NUMBER: DOCUMENT NUMBER:

1995:629937 CAPLUS

TITLE:

123:156260

INVENTOR (S):

Silver halide photographic material with greatly improved residual color on super rapid processing Yamada, Taketoshi; Oonishi, Akira; Usagawa, Yasushi Konishiroku Photo Ind, Japan

PATENT ASSIGNEE(S):

SOURCE:

Jpn. Kokai Tokkyo Koho, 50 pp.

CODEN: JKXXAF

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
		-		
JP 06317878	A2	19941115	JP 1993-108410	19930510
PRIORITY APPLN. INFO.:			JP 1993-108410	19930510

L3 ANSWER 4 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

AB In situ radiolysis of hydroxypyridones in aqueous solution produces several radicals detected by ESR. These are produced by primary and secondary reactions of hydroxyl radicals. Azaquinoidal structures were detected from 3- and 5-hydroxypyridones.

IT 59273-22-2P 129223-43-4P 129223-44-5P

129223-45-6P

RL: PRP (Properties); FORM (Formation, nonpreparative); PREP (Preparation) (formation and ESR of)

RN 59273-22-2 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, ion(1-), radical ion(1-) (9CI) (CA INDEX NAME)

RN 129223-43-4 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, radical ion(1-) (9CI) (CA INDEX NAME)

RN 129223-44-5 CAPLUS

CN 4-Pyridinecarboxylic acid, 1,2,3,6-tetrahydro-2,3,6-trioxo-, ion(2-), radical ion(1-) (9CI) (CA INDEX NAME)

RN 129223-45-6 CAPLUS

CN 4-Pyridinecarboxylic acid, 1,2,3,6-tetrahydro-2,3,6-trioxo-, ion(1-), radical ion(1-) (9CI) (CA INDEX NAME)

ACCESSION NUMBER:

1990:514498 CAPLUS

DOCUMENT NUMBER:

113:114498

TITLE:

In-situ radiolysis ESR studies of hydroxypyridones

AUTHOR(S): Icli, Siddik

CORPORATE SOURCE: SOURCE:

Dep. Chem., Ege Univ., Izmir, Turk. Tetrahedron (1990), 46(8), 2891-902

CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE:

Journal

LANGUAGE:

English

L3 ANSWER 5 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN GI

AB A process for reproducing a Ag image in a layer of photog. material comprises contacting the Ag image with an aqueous acid solution of a compound which

T

II

can be reduced by the Ag to form a reducing agent, in the presence of a Ag complexing agent, causing an imagewise diffusion of the reducing agent to a layer which comprises an azamethine dye I (R = substituent; n = 0-3; R1= NH2, substituted NH2, OH; X, X1, X2 and the 2 C atoms to which X and X1 are joined = optionally substituted coupler moiety). Preferred I are hydroxypyridone dyes II [R = H, optionally substituted (o.s.) alkyl,aralkyl, cycloalkyl, aryl, heterocyclyl, o.s. NH2; R1 = H, CN, CO2R8, CONR8R9, SO3H, SO3-, COR8; R2 = H, OH, CN, CO2R10, CONR10R11, COR10 (R8, R9, R10, R11 = H, o.s. alkyl, aralkyl, cycloalkyl, aryl, heterocyclyl); R3, R4, R5 = H, halo, o.s. alkyl or cycloalkyl, alkoxy; R6, R7 = H, o.s. alkyl, aralkyl, cycloalkyl, aryl, heterocyclyl; NR6R7 = 5- or 6-membered heterocycle; R4R6N and NR6R7 form 2 N-containing heterocyclic rings]. The process is used to reproduce an image in an old photograph on a new support. Thus, an assembly was prepared which comprised a $150-\mu$ -thick transparent cellulose triacetate film support coated with 10 mg compound II (R = Et, R1 = CN, R2 = R6 = R7 = Me, R3 = R4 = R5 = H)/dm2 dispersed in 30

mg gelatin/dm2. A strip of this assembly and a strip of a Ag image print of a bar chart (0.11-10 line pairs/mm) were immersed 10 s in 100 mL 0.25N HCl containing 10 mg pyridinetrione III as auxiliary catalyst, dissolved in 0.5 mL EtO(CH2)2OH. The coatings were then contacted by passing them face-to-face through a pair of rubber rollers at 5 ft/min. After 1 min, there was total bleaching in the areas corresponding to Ag, and the response at 10 lines/mm was 88%.

IT 13445-17-5D, derivs.

RL: CAT (Catalyst use); USES (Uses)

(catalysts, for use in reproduction of old photographs on new support bases)

RN 13445-17-5 CAPLUS

CN 2,3,6(1H)-Pyridinetrione (9CI) (CA INDEX NAME)

ACCESSION NUMBER:

1981:470987 CAPLUS

DOCUMENT NUMBER:

95:70987

TITLE:

Reproduction of photographic material using coupled

hydroxypyridone azamethine dyes

INVENTOR(S):

Wood, Glenn Peter; Long, William Edward; Thomas,

Patrick David Pryce

PATENT ASSIGNEE(S):

Ciba-Geigy A.-G., Switz. Brit. UK Pat. Appl., 14 pp.

SOURCE:

CODEN: BAXXDU

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 2047419	Α	19801126	GB 1980-9867	19800324
GB 2047419	B2	19830518		
PRIORITY APPLN. INFO.:			GB 1979-10540 A	19790326

L3 ANSWER 6 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

The oxidation of hydroxy-2-pyridones by one-electron oxidants was studied and intermediate free radicals observed by ESR spectroscopy. In alkaline media azasemiquinones arising from electron transfer, solvation, and oxidative coupling processes were detected. ESR hyperfine splittings are assigned with the aid of Me substitution and spin ds. explained by considering N perturbation. In acid solution N-protonation produces structural changes in the semiquinone nucleus which affect spin d. distributions. The apparent lifetimes of the radical species can be correlated with their expected tendencies to direct free radical dimerization.

IT 59273-22-2 59273-23-3 59273-24-4

RL: PRP (Properties)

(ESR hyperfine splitting consts. of)

RN 59273-22-2 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, ion(1-), radical ion(1-) (9CI) (CA INDEX NAME)

RN 59273-23-3 CAPLUS

CN 2,3-Pyridinedione, 6-hydroxy-4-methyl-, ion(1-), radical ion(1-) (9CI) (CA INDEX NAME)

RN 59273-24-4 CAPLUS

CN 2,3-Pyridinedione, 6-hydroxy-5-methyl-, ion(1-), radical ion(1-) (9CI) (CA INDEX NAME)

ACCESSION NUMBER:

1976:134745 CAPLUS

DOCUMENT NUMBER:

84:134745

TITLE:

Electron spin resonance studies of azasemiquinone free

radical intermediates in the oxidation of

hydroxypyridones

AUTHOR (S):

Ashworth, P.

CORPORATE SOURCE:

Dep. Chem., Univ. York, York, UK Tetrahedron (1976), 32(2), 261-7

SOURCE:

CODEN: TETRAB; ISSN: 0040-4020

DOCUMENT TYPE:

LANGUAGE:

Journal English

L3 ANSWER 7 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

GI For diagram(s), see printed CA Issue.

The pyridine I (R = H) was halogenated with HBr and HCl to give I (R = Br, Cl). I (R = Cl). HCl was hydrolyzed with HCl to give II. I and COC12 gave III. I and ClCN gave IV.

IT 57892-51-0P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction with thionyl chloride)

RN 57892-51-0 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, 4,5-dichloro- (9CI) (CA INDEX NAME)

ACCESSION NUMBER:

1976:43770 CAPLUS

DOCUMENT NUMBER:

84:43770

TITLE:

Trihalogenated aminopyridinols

AUTHOR (S):

Alt, K. O.; Christen, Edgar; Weis, Claus D.

CORPORATE SOURCE:

Dyestuffs Chem. Dep., Ciba-Geigy Corp., Basel, Switz.

SOURCE:

Journal of Heterocyclic Chemistry (1975), 12(4), 775-8

CODEN: JHTCAD; ISSN: 0022-152X

DOCUMENT TYPE:

Journal English

LANGUAGE:
OTHER SOURCE(S):

CASREACT 84:43770

L3 ANSWER 8 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

AB 4-methyl-2,3,6-trihydroxypyridine, 5-methyl-2,3,6-trihydroxypyridine (I), and 4,5-dimethyl-2,3,6-trihydroxypyridine were prepared I was obtained from 3-hydroxy-6-methyl-2-aza-1,4-benzoquinone (II); the other 2 compds., by literature methods. Their uv and N.M.R. spectra are reported. They undergo CH OH-tautomerism. The compds. undergo rapid autoxidn. to the corresponding azaquinones. Autoxidn. of I leads to II in quant. yield.

IT 19365-33-4P

RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)

RN 19365-33-4 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, 4,5-dimethyl- (8CI) (CA INDEX NAME)

ACCESSION NUMBER:

1968:467192 CAPLUS

DOCUMENT NUMBER:

69:67192

TITLE:

Methyl-substituted 2,3,6-trihydroxypyridines and their

oxidation products

AUTHOR (S):

Knackmuss, Hans Joachim

CORPORATE SOURCE:

Max-Planck-Inst. Med. Forsch., Heidelberg, Fed. Rep.

Ger.

SOURCE:

Chemische Berichte (1968), 101(8), 2679-89

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE:

LANGUAGE:

Journal German

,

L3 ANSWER 9 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN GI For diagram(s), see printed CA Issue.

The bromate oxidation of 5-amino-3-methyl-2-pyridone gave 3-hydroxy-6-methyl-2-aza-1,4-benzoquinone 4-(2,6-dihydroxy-5-methylpyridyl-3-imine) (I). Treating I with concentrated HNO3 gave 3-hydroxy-6-methyl-2-aza-1,4-benzoquinone. Hydrogenation and oxidation gave 4,4'-dihydroxy-5,5'-dimethyl-3,3'-diaza-2,2'-diphenoquinone (II).

IT 17999-44-9P 28518-43-6P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 17999-44-9 CAPLUS

CN 2,3,6(1H)-Pyridinetrione, 5-methyl- (9CI) (CA INDEX NAME)

RN 28518-43-6 CAPLUS

CN Glutaconimide, 2-methyl-4-oxo-, mono(phenylhydrazone) (8CI) (CA INDEX

NAME)

CM 1

CRN 17999-44-9 CMF C6 H5 N O3

CM 2

CRN 100-63-0 CMF C6 H8 N2

 $H_2N-NH-Ph$

ACCESSION NUMBER:

1968:104928 CAPLUS

DOCUMENT NUMBER:

68:104928

TITLE:

Structure and properties of the oxidation product of

5-amino-3-methyl-2-pyridone

AUTHOR (S):

Knackmuss, Hans J.

CORPORATE SOURCE:

Max-Planck-Inst. Med. Forsch., Heidelberg, Fed. Rep.

Ger.

SOURCE:

Chemische Berichte (1968), 101(4), 1148-53

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE:

LANGUAGE:

Journal German

L3 ANSWER 10 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

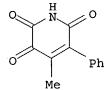
GI For diagram(s), see printed CA Issue.

cf. preceding abstract The unsatd. acyldiazabicyclic ketones (I and II) are converted by heating in methanol to the acylpyrrolinones (III) and 6-acylamidopyridines (IV). In aqueous base I and its methoxy analog give (V) and its 6-methoxy analog, resp., which are readily converted to the pyridone (VI). The benzoyl ketone II in aqueous base gives predominately enamino ketone AcCPh:CHNH2 (VII), with a small amount of VI. Mechanisms for these reactions and for the thermal conversion of II to the uretidine (VIII) are proposed. Initial fragmentation of I and II is suggested to give the azetinone (IX) which is then attacked by water or methanol to give intermediates that undergo cyclizations or fragmentations leading to III, IV, V, and VII. Recyclization of IX leads to VIII.

IT 10137-16-3P

RN 10137-16-3 CAPLUS

CN 2,3-Pyridinedione, 6-hydroxy-4-methyl-5-phenyl- (6CI, 8CI) (CA INDEX



ACCESSION NUMBER:

1967:403026 CAPLUS

DOCUMENT NUMBER:

67:3026

TITLE:

Heterocyclic studies. XXV. Rearrangements of 2-acyl-1,2-diazabicyclo[3.2.0]-3-hepten-6-ones in

methanol and in base

AUTHOR(S):

Moore, James Alexander; Wineholt, Robert L.; Marascia, Frank J.; Medeiros, Robert W.; Creegan, Francis J.

Univ. of Delaware, Newark, DE, USA

CORPORATE SOURCE:

SOURCE:

Journal of Organic Chemistry (1967), 32(5), 1353-60

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE:

Journal English

LANGUAGE:

OTHER SOURCE(S):

CASREACT 67:3026

ANSWER 11 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN L3

2-Amino-5-nitropyridine (10 g.) and 22 g. BzCl in 100 cc. C5H5N warmed 45 AB min., poured into aqueous Na2CO3, and steam distilled, and the distillation residue

filtered gave 17 g. 2-benzamido-5-nitropyridine, tan needles, m. 167°; a 10-g. portion in 180 cc. glacial AcOH hydrogenated 15 min. over 500 mg. 10% Pd-C, treated with 7.2 cc. concentrated HCl, and filtered, the residue extracted with EtOH, and the extract (75 cc.) diluted with 750 cc. Et2O gave 7.5 g. 5-amino-2-benzamidopyridine-2HCl (I.2HCl), m. 220-30°; I.2HCl neutralized with aqueous NaHCO3 gave I, m. 141-2° (H2O containing a trace of Na2S2O4). I.2HCl (1.0 g.) in 5 cc. 20% HCl treated at 0° with 0.28 g. NaNO2 in 3 cc. H2O, kept 15 min. at 0°, treated with urea, added during 0.5 hr. at 95° to 50 cc. 20% HCl, basified, treated with BzCl, and filtered, and the residue treated in H2O with C and added to 5N HCl gave 2-benzamido-5-hydroxypyridine-HCl (II.HCl), m. 215-20°, which with NaHCO3 yielded 425 mg. II, m. 181-2° (MeOH), pK'A 2.6, 8.8 (50% MeOH). II (100 mg.) in 5 cc. aqueous K2CO3 shaken with 3 drops BzCl yielded 130 mg. 3-benzoate (III) of II, needles, m. 148-9° (MeOH). III hydrolyzed with KOH-MeOH gave 85% II, m. 180-2°. II (3.0 q.) in 10 cc. EtOH containing 0.015 mole NaOEt refluxed 2.5 hrs. with 0.93 cc. EtBr and evaporated in vacuo, the solid residue dissolved in aqueous K2CO3, and the product isolated with Et2O gave 900 mg. 5-EtO analog (IV) of II; the aqueous solution neutralized yielded 1.75

g.

unchanged II; the crude IV refluxed 2 hrs. with concentrated HCl, washed with Et20, and evaporated gave 350 mg. (crude) 2-amino-5-ethoxypyridine-2HCl (V.2HCl), m. 143-5°, which with NaHCO3 in the presence of a trace of Na2S2O4 yielded V, m. 47-8°; picrate, yellow needles, m. 240-1°. V (10 mg.) refluxed with Ac20 and evaporated gave 8 mg. 2-AcNH analog of V, needles, m. 108-9° (EtOH). 3-Hydroxypyridine (VI) and 11.2 g. KOH in 500 cc. H2O treated simultaneously with 0.2 mole p-O2NC6H4N2Cl (VII) and 58 g. KOH in 1 l. H2O during 20 min., stirred 1 hr., treated with 50 cc. glacial AcOH, and filtered, and the crude coupling product (44.6 g.) fractionally crystallized from EtOH gave 3 fractions of 5-hydroxy-2-(p-nitrobenzeneazo)pyridine (VIII), violet needles, m. 213-30°; red needles, m. 231-2°; orange needles, m. 214-26°; the orange form recrystd. from EtOH gave orange-red needles, m. 231-2°. VIII (25 g.) in 150 cc. glacial AcOH hydrogenated 0.5 hr. at 45 lb. over 0.3 g. 10% Pd-C, the mixture treated under N with 48 cc. 48% HBr and filtered, the residue washed with AcOH, the combined AcOH solns. evaporated in vacuo, the residue dissolved in H2O,

basified with Na2CO3, shaken with 34 g. BzCl in the presence of a trace of SnCl2, and filtered, the residue hydrolyzed with KOH-MeOH, the mixture concentrated, acidified with HCl, and filtered, and the residue washed with

Et20

gave 20.5 g. II.HCl, m. 215-20°. VIII (2.5 g.) in 250 cc. concentrated HCl and 150 cc. MeOH treated with 25 g. SnCl2 in small portions, warmed on the steam bath, basified with 50% aqueous KOH, and evaporated, the residue dissolved in 25 cc. iced H2O, treated with sufficient 50% aqueous KOH to dissolve the Sn salts, and shaken with 10 cc. BzCl at 0-5°, and the organic phase washed with H2O, kept overnight with 10% aqueous Na2CO3, continuously extracted with MeOH and filtered yielded 2.30 g. 2-dibenzamido-5-benzoyloxypyridine benzoate, needles, m. 182-3° (MeOH). VII from 1.38 g. p-O2NC6H4NH2 neutralized with NaOAc, treated at 25° with 0.85 g. VI in 50 cc. H2O, kept overnight, and filtered gave 1.78 g. crude product; a 31-mg. sample in 2 cc. EtOH chromatographed on 3 g. Al203 yielded 5.5 mg. 3-hydroxy-2-(p-nitrobenzeneazo)pyridine (IX), red needles, m. 234-5° (MeOH); the chromatogram of a similar run in alkaline solution gave 13.4% IX. IX (40 mg.) reduced and treated in aqueous

NaHCO3 with BzCl gave the N,N,O-tri-Bz derivative (X) of 2-amino-3hydroxypyridine (XI). XI.HBr (500 mg.) in aqueous NaHCO3 shaken with 1 cc. BzCl yielded 900 mg. X, needles, m. 169-70° (MeOH). X hydrolyzed with KOH-MeOH yielded the mono-Bz derivative of XI, needles, m. 95-6°; picrate, yellow needles, m. 237-8° (EtOH). II (7.0 g.) in 20 cc. 48% HBr refluxed 3 hrs. and concentrated in vacuo, the sirupy residue dissolved in a small volume of EtOH and diluted with Et2O gave 5.7 g. 2-amino-5-hydroxypyridine-HBr (XII.HBr), m. 120-5°. XII.HBr dissolved in aqueous NaHCO3 containing a few mg. Na2S2O4 and the product

isolated

with Et20 gave XII, needles, m. 116-17° (MeOH-C6H6); picrate, yellow needles, m. 225-7° (decomposition). II hydrolyzed with concentrated HCl yielded XII.HCl, needles, m. 125-6° (EtOH-Et2O). XII.HBr in 20 cc. 20% H2SO4 treated with cooling and stirring with 4.5 g. NaNO2 in 5 cc. H2O gave 1.75 g. 3,6-dihydroxy-2-nitrosopyridine (XIII), bright red needles, decomposing 210° (H2O), pKA 8.4 (H2O). XII.HBr (500 mg.) treated with 3 cc. cold concentrated H2SO4 and then with 170 mg. NaNO2, stirred 10 min., heated, cooled, poured onto ice, neutralized with solid NaHCO3, and treated with 1 cc. BzCl, and the product isolated with Et2O yielded 550 mg. 2,5-dihydroxypyridine benzoate (XIV), needles, m. 187-9° (C6H6). XIV (45 mg.) in 1 cc. 48% HBr refluxed 0.5 hr., cooled, diluted with H2O, washed with Et2O, neutralized with NaHCO3, and extracted with 1:1 C6H6-EtOH, the extract distilled, and the resulting pale yellow glass crystallized

from EtOH yielded 15 mg. 2,5-dihydroxypyridine (XV), needles, m. 245-7° (EtOH). XIV (100 mg.) hydrolyzed with HBr and evaporated, the residual crude XV.HBr dissolved in 20% H2SO4, and the solution treated at 15-20° with 75 mg. NaNO2 gave XIII. XIII (200 mg.) in 4 cc. concentrated HCl and 4 cc. EtOH treated with 500 mg. SnCl2, warmed on the water bath, and concentrated to half-volume gave 190 mg. 2-amino-3,6-dihydroxypyridine-HCl (XVI.HCl), pale yellow needles; it darkened rapidly in air. A sample of XVI.HCl added to aqueous NaHCO3 gave a brilliant indigo precipitate from a deep blue

solution XVI.HCl (15 mg.) in 2 cc. C5H5N treated with 3 drops BzCl, warmed briefly, poured into iced HCl, and filtered gave 20 mg. 2-benzamido-3-benzoyloxy-6-pyridone, needles, m. 243-4° (CHCl3-EtOH). 3-Hydroxy-4-methyl-5-phenylpyridine (3.2 g.) in 100 cc. H2O containing 1 equivalent NaOH treated with 0.017 mole VII, stirred 1 hr., acidified, and filtered, the residue (6.83 g.) extracted with C6H6 in a Soxhlet apparatus, and the insol. residue (3.9 g.) recrystd. from EtOH gave 5-hydroxy-3-phenyl-4-methyl-2-(p-nitrobenzeneazo)pyridine (XVII), stout red needles, m. 261-9° (decomposition) (EtOH); the extract evaporated yielded 3-hydroxy-4-methyl-5-phenyl-2-(p-nitrobenzeneazo)pyridine (XVIII), golden-red laths, m. 230-5°. XVIII (1.77 g.) in 150 cc. AcOH hydrogenated over 0.25 g. 10% Pd-C, treated with HBr, and filtered, the

residue extracted with H2O, and the extract treated with NaOH and BzCl gave

1.83 g. p-C6H4(NHBz)2; the AcOH filtrate evaporated, the residue treated with NaOH and BzCl, and the product isolated with Et2O gave 2.80 g. N,N,O-tri-Bz derivative (XIX) of 2-amino-3-hydroxy-4-methyl-5-phenylpyridine (XX), needles, m. 182°. XIX (250 mg.) and 500 mg. KOH in 6 cc. 80% MeOH refluxed 0.5 hr., the MeOH evaporated, the residue diluted with H2O, acidified with dilute acid, and neutralized with NaHCO3, and the precipitate treated with picric acid gave the picrate of the Bz derivative (XXI), of XX, m. 213°. XIX (280

mg.) in 5 cc. 48% HBr refluxed 1 hr., cooled, washed with Et2O, and neutralized with NaHCO3 gave 125 mg. (crude) XX, needles, m. 210° (decomposition), pKA 6.05, 9.9 (50% MeOH); picrate, golden needles, m. 260° (decomposition) (EtOH). XX with BzCl in C5H5N gave the 2-benzamido-5-benzoate ester, needles, m. 195-6°. XX (25 mg.) and 50 mg. recrystd. picryl chloride heated 10 min. on the water bath, cooled, and poured into H2O gave 20 mg. 7,9-dinitro-4-methyl-3-phenyl-10pyrido[3.2-b] [1.4]benzoxazine, red prisms, m. 196° (MeOH). XVII (1.94 g.) hydrogenated in the usual manner and the crude aminohydroxypyridine benzoylated gave a noncryst. polybenzoyl derivative which treated directly with KOH-MeOH yielded the N-Bz derivative (XXII) of 2-amino-5-hydroxy-4-methyl-3-phenylpyridine (XXIII), needles, m. 216-17° (CHCl3-Et20). XXII (325 mg.) in 3 cc. concentrated H2SO4 warmed 10 min., poured onto ice, washed with Et2O, neutralized with NaHCO3, and filtered gave 205 mg. (crude) XXIII, rods, m. 190-5° (decomposition), pKA 6.05, 10.25 (50% MeOH). XXII in Et2O treated with C5H5N and BzCl and evaporated, and the oily residue triturated with aqueous NaHCO3 yielded the 2-benzamido-5-benzoate ester (XXIV) of XXIII, needles, m. 199-200° (EtOAc). XXIII benzoylated in the usual manner and then hydrolyzed with KOH-MeOH gave XXIII. XXIII (100 mg.) in 2.5 cc. 60% H2SO4 treated at -5° with 35 mg. NaNO2, stirred at 0° until the gas evolution ceased, warmed to 50°, cooled, neutralized with K2CO3, and filtered yielded 60 mg. 2,5-dihydroxy-4-methyl-3-phenylpyridine (XXV), needles, m. 250-60° (MeOH). XXIII (850 mg.) in 20 cc. 20% H2SO4 treated at room temperature with 1.20 g. NaNO2 in 7 cc. H2O, stirred 10 min., and filtered gave 650 mg. 6-NO derivative (XXVI) of XXV, golden-red prisms, m. 250-3° (EtOH), pkA below 2, 8.65 (50% MeOH); the mother liquor from the crude XXVI extracted with Et20 yielded 70 mg. 2-hydroxy-4-methyl-5-phenyl-1-azaquinone (XXVII), cream-colored needles; the Et20-extracted aqueous acid solution

neutralized with solid NaHCO3 yielded 110 mg. (crude) 6-NO derivative of XXIII, yellow needles, m. above 280° (Me2CO). XXVI (150 mg.) in 2.5 cc. 40% H2SO4 heated on the steam bath to solution, cooled, and neutralized with solid NaHCO3, and the product isolated with Et20 gave 60 mg. XXVII, pale yellow needles, m. 160-1° (H2O); the EO value for the reaction was -0.40 v. against a calomel electrode. XXV (10 mg.) in 0.5 cc. 20% H2SO4 treated with 20 mg. NaNO2 and kept 2 days at room temperature yielded 5.5 mg. XXVII, m. 159-60°. XXVI (250 mg.) in 7 cc. 40% H2SO4 refluxed 1 hr. and steam distilled, and the product isolated from the distillate with Et20 gave 75 mg. 3-methyl-4-phenylmaleic anhydride (XXVIII), needles, m. 95-6° (sublimed). XXVII (21 mg.) and 10.2 mg. o-C6H4(NH2)2 in 2 cc. AcOH warmed 1 hr. on the water bath and evaporated, and the residue treated with EtOH gave 2-hydroxy-4-methyl-3phenylpyrido[2.3-b]-quinoxaline, pale yellow needles, m. 275° (AcOH). XXVII (50 mg.) in 3 cc. Ac2O heated 1 hr. at 75° with 500 mg. Zn dust, filtered, and evaporated gave 55 mg. 2,3,6-trihydroxy-4-methyl-5phenylpyridine triacetate, prisms, m. 106-7° (Et20-hexane).

IT10137-16-3, 2,3-Pyridinedione, 6-hydroxy-4-methyl-5-phenyl-(preparation of)

10137-16-3 CAPLUS

RN

2,3-Pyridinedione, 6-hydroxy-4-methyl-5-phenyl- (6CI, 8CI) (CA INDEX

ACCESSION NUMBER:

1960:34227 CAPLUS

DOCUMENT NUMBER:

54:34227

ORIGINAL REFERENCE NO.:

54:6695f-i,6696a-i,6697a-h

Heterocyclic studies. VII. The preparation and

reactions of 2-amino-5-hydroxypyridines; the formation

of an azaquinone

AUTHOR (S):

Moore, James A.; Marascia, Frank J.

CORPORATE SOURCE:

Univ. of Delaware, Newark

SOURCE:

Journal of the American Chemical Society (1959), 81,

6049-56

CODEN: JACSAT; ISSN: 0002-7863

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Journal

LANGUAGE:

Unavailable

 L_3 ANSWER 12 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN

2-Hydroxy-5-aminopyridine (I) HCl salt (3.0 g.) in 25 cc. conductivity H2SO4 AB warmed gently until the HCl evolution ceased, cooled, poured onto 50 g. ice, treated rapidly with stirring with 1.1 g. KBrO3 in 25 cc. H2O below 5°, kept several hrs. at 3-5°, and filtered, the residue washed with ice H2O, dried, extracted 1 week with absolute EtOH in a Soxhlet extractor, and the insol. residue recrystd. from HCONMe2 and molten AcNH2 gave 0.80 g. 1-aza-6-hydroxy-2,5-benzoquinone (II), purple-brown microcrystals with a metallic sheen, decompose above 300°

(indefinite); the EtOH extract concentrated and cooled gave 0.40 g.

purple-brown

rhombs, decompose above 300°, which appeared to be identical with the insol. material except for the solubility properties; both fractions dissolved in alkali with an intense blue color which faded rapidly with absorption of O and evolution of NH3; the alkaline solution finally turned red-yellow and fluoresced in ultraviolet light. 2-Hydroxy-3-aminopyridine (III).HCl (1.0 g.) in 10 cc. concentrated H2SO4 warmed until the evolution of HCl ceased, treated at -20° with 0.3 g. CrO3 in 5 cc. concentrated H2SO4 with stirring, kept 2 hrs. at -20°, warmed slowly at room temperature, poured onto ice, and filtered, and the purple precipitate (0.25 g.) extracted in the usual

manner with EtOH gave a soluble and an insol. fraction of II. II (soluble or insol. fraction) (0.1 g.) and 0.5 g. Zn dust heated with 3 cc. Ac20 on the steam bath, the pale yellow, pale blue fluorescing solution filtered hot and evaporated in vacuo, and the residue recrystd. from EtOH gave 0.15 g. 2,3,6-triacetoxypyridine, pale cream platelets, m. 159° (from EtOH). 2,3-Dihydroxypyridine oxidized with MnO2 or with KBrO3 in the usual manner gave soluble and insol. II and a trace of an unidentified light yellow solid, m. above 300° (decomposition). 3-Amino-4-hydroxypyridine HCl salt oxidized in the usual manner gave only intractable red tars, even with insufficient amts. of oxidant at -50° 3-Nitro-4-chloropyridine (3.2 g.) and 2.6 g. NaN3 in 23 cc.MeOH and 2 cc. H2O warmed 10 min. at 35-40°, filtered, concentrated to 1/2 the original volume, and cooled gave 2.55 g. 3-nitro-4-azidopyridine, pale yellow rods, m. 89° (decomposition); it decomposed when heated a few sec. above 90° with a violet gas evolution, and formed a yellow oil which changed rapidly into a dark insol. residue, did not melt or decompose below 300°. 2-Amino-5-methylpyridine nitrated, diazotized, and hydrolyzed gave 38% 2-hydroxy-3-nitro-5-methylpyridine (IV), m. 253-5°. IV (30.8 g.) in 400 cc. 2% AcOH warmed on the H2O bath with occasional swirling with excess Fe filings, neutralized with CaCO3, and filtered hot, the residue

washed with the hot H2O, the combined filtrate treated with stirring and cooling with excess Ac2O and filtered and the filter residue recrystd. from EtOH gave 22.2 g. 2-hydroxy-3-acetamido-5-methylpyridine (V), leaflets, m. 253° with a change to needles at about 220°. 2-Amino-3-methylpyridine nitrated, diazotized, and hydrolyzed yielded 71% 2-hydroxy-3-methyl-5-nitropyridine which reduced and acetylated in the usual manner gave 69% 2-hydroxy-3-methyl-5-acetamidopyridine (VI), needles, m. 247° (from EtOH). V (3.0 g.) in 50 cc. 16% H2SO4 heated 5 min. at 95-100°, cooled to 25°, treated with 1.0 g. KBrO3 in 25 cc. H2O, kept below 40°, then 2 hrs. at room temperature, refrigerated 24 hrs., and filtered, and the residue (0.7-1.0 g.) washed with cold H2O, dried, and recrystd. from MeOH gave impure quinhydrone (VII) of 3-aza-4-hydroxy-5-methyl-1,2-benzoquinone, birefringent green-gray leaflets, decompose above 300° (indefinite); it dissolved in alkali with an intense blue color which changed after a few hrs. to yellow-red. VII (2.0 g.) and 5.0 cc. PhNHNH2 in the min. amount of 10% AcOH heated 6 hrs. on the H2O bath and cooled gave 1.2 g. monophenylhydrazone, brown-red needles, m. 254° (decomposition), changing to yellow-red at 210°. VI oxidized in the usual manner gave the quinhydrone, green-gray platelets, decompose above 300°; deep purple in dioxane changing to red-yellow with the simultaneous formation of a green-blue fluorescence to ultraviolet light; soluble in alkali with decomposition and green-blue fluorescence.

RN 13445-17-5 CAPLUS

CN 2,3,6(1H)-Pyridinetrione (9CI) (CA INDEX NAME)

RN 17999-44-9 CAPLUS CN 2,3,6(1H)-Pyridinetrione, 5-methyl- (9CI) (CA INDEX NAME)

O N O Me

ACCESSION NUMBER: 1957:90711 CAPLUS

DOCUMENT NUMBER: 51:90711

ORIGINAL REFERENCE NO.: 51:16458d-i,16459a-c

TITLE: Azaquinones. I. Oxidation of certain hydroxy- and

aminopyridines

AUTHOR(S): Boyer, J. H.; Kruger, S.

CORPORATE SOURCE: Tulane Univ., New Orleans, LA

SOURCE: Journal of the American Chemical Society (1957), 79,

3552-4

CODEN: JACSAT; ISSN: 0002-7863

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LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 51:90711

ANSWER 3 OF 12 CAPLUS COPYRIGHT 2004 ACS on STN ACCESSION NUMBER: 1995:629937 CAPLUS

DOCUMENT NUMBER: 123:156260

ENTRY DATE: Entered STN: 22 Jun 1995

Silver halide photographic material with greatly TITLE: improved residual color on super rapid processing INVENTOR(S): Yamada, Taketoshi; Oonishi, Akira; Usagawa, Yasushi

PATENT ASSIGNEE(S): Konishiroku Photo Ind, Japan SOURCE: Jpn. Kokai Tokkyo Koho, 50 pp.

Patent

CODEN: JKXXAF DOCUMENT TYPE:

LANGUAGE:

Japanese INT. PATENT CLASSIF.:

MAIN: G03C001-83 SECONDARY: C09K003-00

CLASSIFICATION: 74-2 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE ---------------JP 06317878 19941115 JP 1993-108410 19930510 PRIORITY APPLN. INFO.: JP 1993-108410 19930510 PATENT CLASSIFICATION CODES:

PATENT NO. CLASS PATENT FAMILY CLASSIFICATION CODES

--------------JP 06317878 ICM G03C001-83

ICS C09K003-00

GRAPHIC IMAGE:

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

ABSTRACT:

The title photog. material has ≥ 1 layer containing compound I-V (X1 = 0, S, SO2, NR1(R1 = H, alkyl, aryl, heterocyclyl); L1-15 = methine; n1-5 = 0-1; Z = 0non-metallic atoms required to form aromatic ring; Y = alkylsulfonyl, arylsulfonyl, aryloxycarbonyl, carbamoyl; R2 = aryl; R3 = R1; when R3 = aryl, R4 = aryloxycarbonyl, acylamino, ureido, carboxy, carbamoyl, cyano, hydroxy, alkoxy, aryloxy, amino, sulfamoyl, sulfone amido; when R3 = H, alkyl, heterocyclyl, R4 = H, alkyl, aryl, heterocyclyl, alkoxycarbonyl or group defined above for R4; R5-7 = organic group; X2-4 = O, S; R8, R9 = H, alkyl, aryl, heterocyclyl; each compound of I-V containing at least 1 of carboxy, sulfone amido, or sulfamoyl).

SUPPL. TERM: photog material specific compd

167014-94-0

INDEX TERM: Photographic films

(containing specific compound)

INDEX TERM: 71620-29-6 160816-96-6 167014-64-4 167014-65-5

167014-66-6 167014-67-7 167014-68-8 167014-69-9 167014-70-2 167014-71-3 167014-72-4 167014-73-5 167014-74-6 167014-75-7 167014-76-8 167014-77-9 167014-78-0 167014-79-1 167014-80-4 167014-81-5

167014-82-6 167014-83-7 167014-84-8 167014-85-9 167014-86-0 167014-87-1 167014-88-2 167014-89-3 167014-90-6 167014-91-7 167014-92-8 167014-93-9

167014-95-1 167014-96-2 167014-97-3 ROLE: DEV (Device component use); USES (Uses)

(contained in photog. material for greatly improving

residual color on super rapid processing)

INDEX TERM: 167015-01-2P

ROLE: SPN (Synthetic preparation); PREP (Preparation)

(prepared for photog. material)

98-01-1, Furfural, reactions 64542-27-4 90721-27-0,

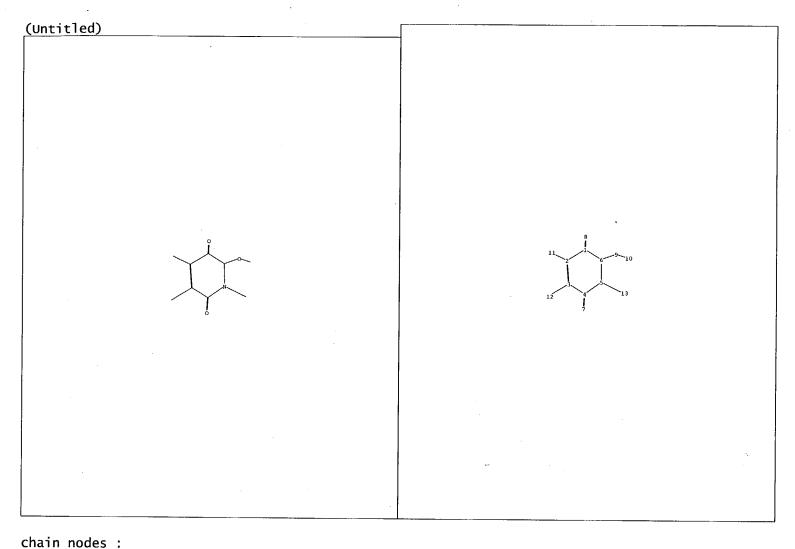
5-Benzofurancarboxylic acid 167014-98-4

167014-99-5 167015-00-1

ROLE: RCT (Reactant); RACT (Reactant or reagent)

(preparing specific compound for photog. material)

INDEX TERM:



7 8 9 10 11 12 13

ring nodes:
 1 2 3 4 5 6

chain bonds:
 1-8 2-11 3-12 4-7 5-13 6-9 9-10

ring bonds:
 1-2 1-6 2-3 3-4 4-5 5-6

exact/norm bonds:
 1-2 1-6 1-8 2-3 3-4 4-5 4-7 5-6 5-13 6-9 9-10

exact bonds:
 2-11 3-12

Match level:
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:CLASS 8:CLASS 9:CLASS 10:CLASS 11:CLASS 12:CLASS 13:CLASS

(J3)